PME RESEARCH LABORATORY, SEPTEMBER 1980

PROJECT TITLE : ANALYTICAL INVESTIGATIONS

PERIOD COVERED : AUGUST 30 - SEPTEMBER 25, 1980

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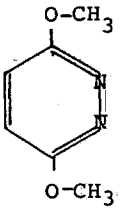
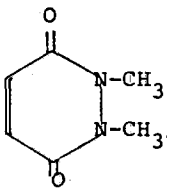
COMPOUND IDENTIFIACTION

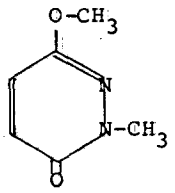
The pesticide laboratory requested the identification of a compound obtained by the reaction of maleic hydrazide with dimethyl sulfate and which was supposed to have structure I or II (1).



I



II



III

The compound of mp. 49-51° (from cyclohexane) and composition $C_6H_8O_2N_2$, gives in GC^2 a single peak at RT 4.5 min (column 12 m x 0.2 mm, fused silica with SP 2100, temp. programme 80° to 150°, 15°/min). GC^2/MS shows M^+ at 140 m/e (100 %) and main fragments at 112 (34), 82 (21), 80 (33), 69 (74) and 54 (20) m/e.

The final assignment of structure is based on ^1H and ^{13}C-NMR. The ^1H-NMR spectrum (60 MHz, DMSO) shows the signals of two different CH_3 protons at 3.5 and 3.7 ppm (1:1) and of the HC= protons at 6.9-7.2 ppm. In the ^{13}C-NMR spectrum ($CDCl_3$) signals are found for $\underline{C}H_3O$ and $\underline{C}H_3N$ at 54.2 and 39 ppm and for two not identical olefinic carbons at 126.3 and 132.3 ppm.

From these data, structure III has been assigned.

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ORGANIC ACIDS

For Candida utilis 707/3 metabolism control Biotechnology requested quantitation of acetic, citric, lactic, malic, succinic, fumaric, tartaric and pyruvic acid (2). The possibilities of a group analysis as p-bromophenacyl derivatives by GC or LC are presently being investigated.

CF-ANALYSIS

- The compound simulating the presence of coumarin in some filler extracts analyzed for CF (3) was identified by GC^2/MS as antioxydants BHT (2,6 di-tert-butyl-4-methylphenol). The compound, as well as diethylphtalate, derived from the GC sample microvial caps used (supplier: Infochroma).

- The GC method has been extended to the analysis of dihydrocoumarin and of 6-methylcoumarin in filler by using n-hexadecane (C_{16}) as internal standard. The chromatogram of a synthetic mixture of coumarin, dihydrocoumarin and of 6-methylcoumarin is given in figure 1.

ROUTINE ANALYSES

Phosphate (165 samples), sulfate (105), amino acids (59) and alcohols (40) were determined for Biotechnology, Process Dev. and Product Research.

Coumarin was analyzed in 30 brands.

REFERENCES

(1) M. Speck, PME Research Lab. Monthly Progress Report, September 1980.

(2) Memo of H. Gaisch to W. Fink, September 1980.

(3) E. Lecoultre, PME Research Lab., Monthly Progress Report, August 1980.

E. Lecoultre

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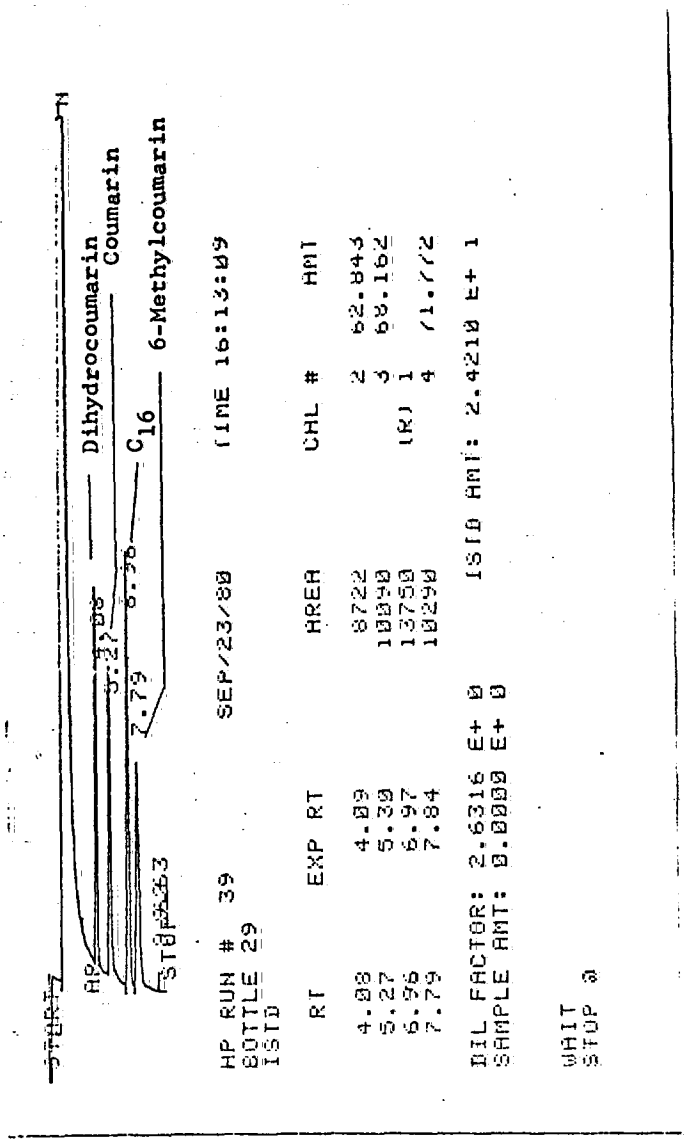
Figure 1 : Gas Chromatogram of a Synthetic Mixture of Dihydrocoumarin, Coumarin and 6-Methylcoumarin.

Internal Standard: n-Hexadecane (C_{16}).



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